Materials Sciences and Applications, **2011, 2, 638-642**
doi:10.4236/msa.2011.26087 Published Online June 2011 (http://www.SciRP.org/journal/msa)

Synthesis and Magnetic Properties of Conventional and Microwave Calcined Strontium Hexaferrite Powder

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Received December 24th, 2010; revised March 21st, 2011; accepted May 17th, 2011.

ABSTRACT

Powders of strontium hexaferrite (*$SrFe_{12}O_{19}$-SrF*) *have been prepared by the sol-gel process. The prepared precursor was calcined in two different calcination techniques*, *using conventional furnace and microwave furnace. Thermal analysis studies showed exothermic and endothermic reaction peak at room temperature to* 1200˚*C. An investigation of $SrFe_{12}O_{19}$ crystalline powder from the structural and magnetic aspect is performed using X-ray diffraction* (*XRD*), *high resolution scanning electron microscopy (HR-SEM) and vibrating sample magnetometer* (*VSM*). *The average particle diagonal size of $SrFe_{12}O_{19}$ powder was* 80 - 100 *nm in conventional and* 40 - 70 *nm in microwave calcinations respectively. XRD result showed the formation of $SrFe_{12}O_{19}$ of the sample calcined at* 900˚*C with Fe*/*Sr*: *D-Fructose ratio* = 12.

***Keywords*:** *Sol-Gel*, *Strontium Hexaferrite*, *D-Fructose*, *Magnetization*

1. Introduction

The M-type Strontium Hexaferrite-$SrFe_{12}O_{19}$ (SrF) is a hard magnetic material due to its high coercivity. $SrFe_{12}O_{19}$ crystallize with 64 ions per unit cell on 11 different symmetry sites, the magnetic structure given by Gorter model, 24 Fe^{3+} atoms are arranged over five distinct sites: three octahedral sites and two tetrahedral sites. These five sites are coupled via ferromagnetic superexchange interaction through O^{2-} ions [1]. Recently, Wang *et al.*, reported a correlation between magnetic properties and particles morphology of $SrFe_{12}O_{19}$ [2]. By controlling the microstructure, morphology and chemical composition and particle size, the magnetic properties of the material can be improved. Ferrites, typically spinel ferrite and magnetoplumbite ferrite, can be used as recording materials, microwave devices, humidity sensors, pigments etc. Compared with spinel ferrite, magnetoplumbite ferrites, strontium ferrite have attracted more scientific research in recent years due to their high uniaxial magnetic anisotropy, high saturation magnetization and high coercivity [3,4]. In order to get homogeneous ferrite, several techniques have been used in the preparation of Sr hexaferrite, such as the chemical co precipitation [5], hydrothermal [6], sol-gel [7,8], micro emulsion [9], and citrate precursor [10] have been developed. Therefore, the preparation of $SrFe_{12}O_{19}$ having high purity, ultrafine size, good dispersion and excellent magnetism has been the focus of recent research [7,11]. The growing interest during the past decade is essentially due to the fact that microwave heat treatment could influence the microstructure can improve the material properties. Conventional furnace heating samples by the surface heating mechanism and depending on the rate of heating, a large thermal gradient from the surface to the centre can be generated within a sample, particularly with materials heaving a poor thermal conductivity. Microwave heating would be promising because it is a self-heating process of absorbing the electromagnetic energy. As a result of the internal and volumetric heating at high heating rate may lead to reduction in manufacturing cost on account of energy savings, shorter processing times and improved product uniformity and yields, limited grain growth throughout the ceramic body. It is well known that the heating rate and thermal efficiency of the microwave heating is higher than those of conventional method [12, 13]. In this paper, we report the preparation of $SrFe_{12}O_{19}$

by sol-gel process and important hysteresis parameters of the powders with influence of convention and microwave calcinations.

2. Experimental

The powder was prepared by sol-gel technique using D-Fructose as the fuel. Analytical grade $Ba(NO_3)_2$, $Fe(NO_3)_3{\cdot}9H_2O$ and D-Fructose were used as starting materials. Nitrate and fuel ratio is 1:1. Stoichiometric amount of metal nitrates and fuel were taken, dissolved in distilled water and stirred by magnetic stirrer for 2 h to get a solution. Sol was heated at 80˚C with stirring continuously, finally it changed in to sticky liquid gel and it was preheated at 130˚C in a hot air oven for two days to get precursor. The precursor was calcined (Conventional and microwave) at 900˚C, to get crystalline barium hexaferrite powder.

2.1. Characterization

Thermo gravimetric analysis of the mixture composed of barium nitrate, iron nitrate and D-Fructose (precursor) were carried out between 28˚C and 1200˚C on NETZSCH STA 409 C/CD in the static air atmosphere at the heating rate of 10˚C per minute. The DTA analyses of same mixtures were also carried out on the same instrument at same condition. The crystalline phases were identified by means of X-ray diffraction (XRD) measurements (PANalytical X'pert pro) CuKα radiation at 45 kV and 40 mA (λ = 0.15406 nm) in a wide range of 2θ ($10^\circ < 2\theta < 80^\circ$). The surface morphology and size of the ferrite particles were studied by using FEI Quanta FEG 200-High resolution scanning electron microscope (HR-SEM) and Magnetization measurements at room temperature were carried out on Lakeshore Vibrating Sample Magnetometer (VSM) at a maximum applied field of 15,000 Gauss at room temperature.

3. Results and Discussion

The thermogram of the precursor of barium hexaferrite derived by mixing of barium nitrate, ferric nitrate and D-Fructose as shown in **Figure 1**. TGA shows the initial weight loss from 28˚C to 185˚C due to the loss of absorbed water [14]. The subsequent loss up to 400˚C is associated mainly to the decomposition of the D-fructose. In order to verify this, separate TGA was undertaken for D-fructose, thermogram is shown in **Figure 2**. It shows the major weight loss between 200˚C and 400˚C thus supporting our assignment. Therefore, D-Fructose provides self heat to promote the reaction and to reduce the crystallization temperature of the hexaferrite. The stage of decomposition between 400˚C and 775˚C is due to decomposition of nitrates and starting formation of hexaferrite. There is no considerable weight loss above

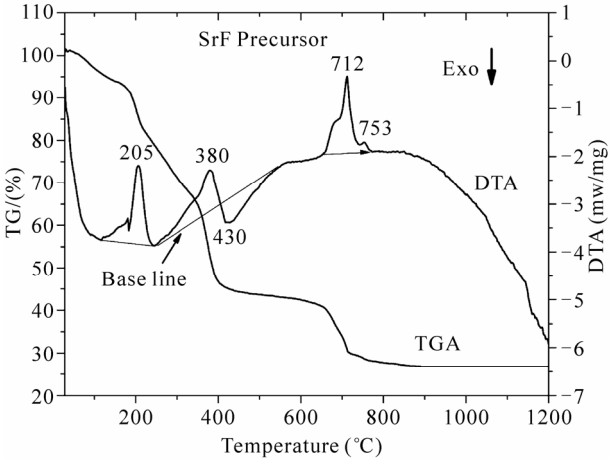

Figure 1. TG-DTA curves for the precursor.

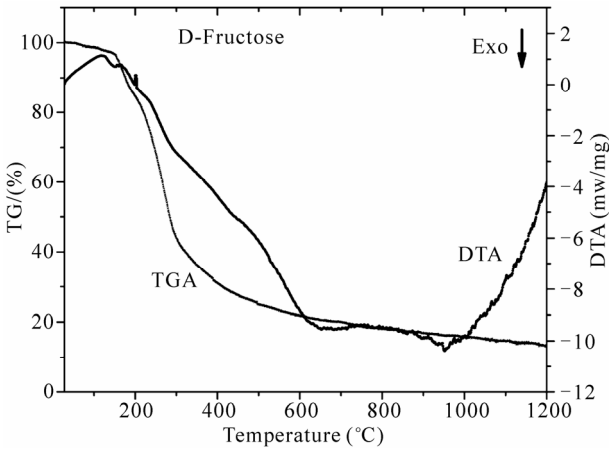

Figure 2. TG-DTA curves for D-fructose.

900˚C, confirming the formation of the stable Strontium hexaferrite this analysis, therefore illustrates the optimum calcinations temperature for Strontium hexaferrite is around 900˚C.

The sequences of reaction taking place is shown in the following steps

$$\text{Precursor} \xrightarrow{200^\circ\text{C} - 500^\circ\text{C}} Fe_2O_3 + SrO$$

$$Fe_2O_3 + SrO \xrightarrow{250^\circ\text{C} - 750^\circ\text{C}} SrFe_2O_4$$

$$5Fe_2O_3 + SrFe_2O_4 \xrightarrow{\text{above } 750^\circ\text{C}} SrFe_{12}O_{19}$$

Figure 3 shows the XRD patterns of the powders conventionally calcined at temperatures 500˚C, 750˚C and 900˚C for 3 h in air, respectively. The precursor is calcined at 500˚C; the powders can be described as Fe_2O_3 and SrO and then the phase of $SrFe_2O_4$ and hexagonal $SrFe_{12}O_{19}$ can been detected for samples calcined at 750˚C. Clear diffraction peak of $SrFe_{12}O_{19}$ can be obtained at 900˚C, which coincides with the JCPDS file

number: 84-1531. Calcination temperature and intermediate Fe_2O_3 plays an important role in the formation of Strontium Hexaferrite. The phase development microwave calcined powder at different temperatures, 500˚C, 750˚C and 900˚C for 10 minutes, the peaks corresponding to the standard diffraction pattern of SrO, $SrFe_2O_4$ and $SrFe_{12}O_{19}$ and is shown in **Figure 4**.

In order to visualize the conventional calcined powder are in elongated hexagonal like structure, diagonal size vary in the range of 80 - 100 nm and it is not well defined shape (**Figure 5**). The HR-SEM micrograph for the microwave calcined powder at 900˚C for 10 minutes is shown in **Figure 6**. The particles are hexagonal platelets and well crystalline strontium ferrite, diameters are in the range of 40 nm to 70 nm. This type of shape is usually observed for $BaFe_{12}O_{19}$ or $SrFe_{12}O_{19}$ obtained by sol-gel process [4,15]. The morphology of the microwave calcined powder samples reveal smaller particles compared

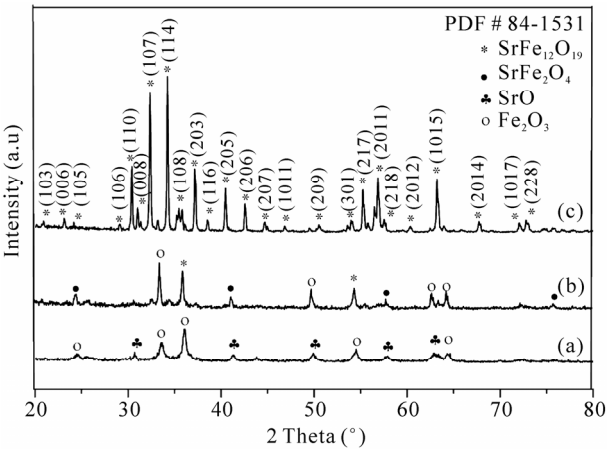

Figure 3. XRD patterns of the powders calcined at different temperatures: (a) 500˚C, (b) 750˚C, and (c) 900˚C for 3 h.

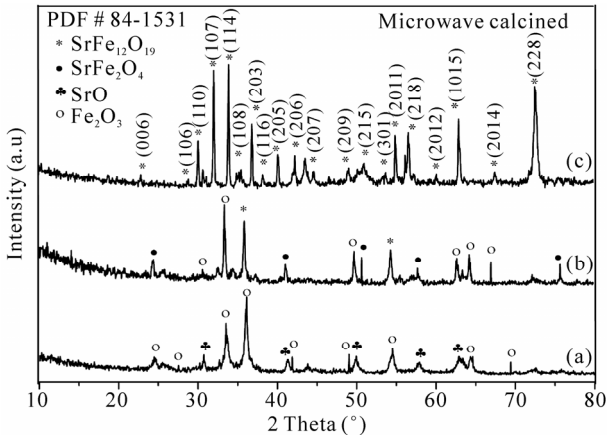

Figure 4. XRD patterns of the powders microwave calcined at different temperatures: (a) 500˚C, (b) 750˚C, and (c) 900˚C for 10 minutes.

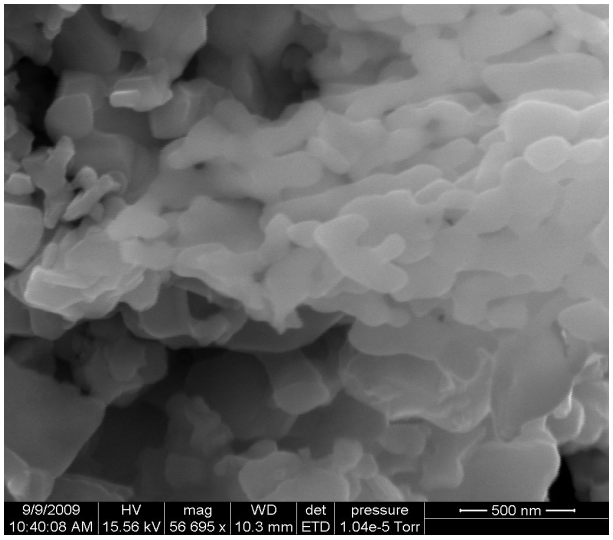

Figure 5. HR-SEM image of conventionally calcined powder at 900˚C for 3 h.

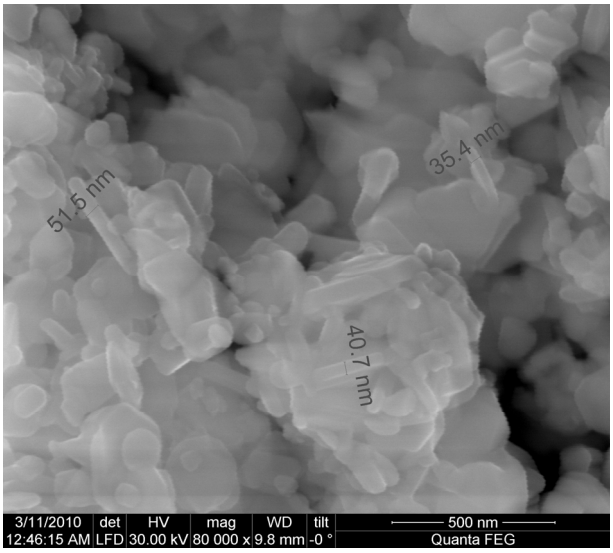

Figure 6. HR-SEM image of microwave calcined powder at 900˚C for 10 minutes.

to the conventionally calcined powder. Hard magnetic materials with hexagonal structure is mainly due to the microwave energy coupled through polarization, electronic and ionic conductivity loss therefore a smaller particle size resulted from the enhanced diffusion and accelerated densification [16].

Figure 7 shows the magnetization versus applied field for conventional and microwave treated samples at room temperature. The reduction in M_s in microwave calcined powder can be attributed to the decrease in the size of the particles. The observed value of saturation magnetization 47 $A \cdot m^2/kg$ for the sample conventionally calcinated at 900˚C are far from the theoretical M_s value of 74.3

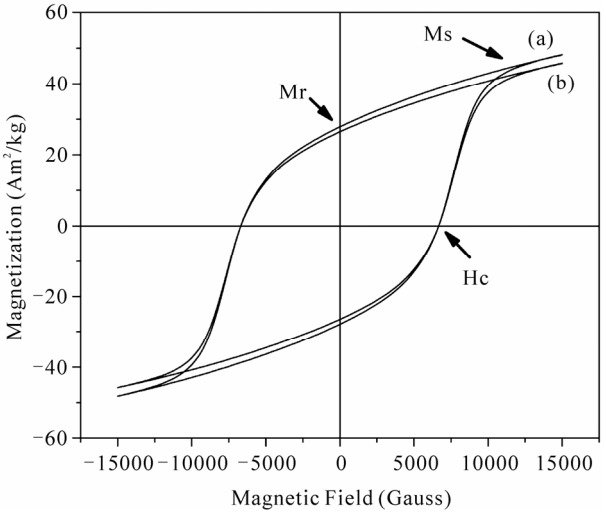

Figure 7. Magnetization curve of $SrFe_{12}O_{19}$ (a) powder conventionally calcined at 900˚C for 3 h; (b) powder microwave calcined at 900˚C for 10 minutes.

$A.m^2/kg$ and the coercivity 6,709 Gauss very close to the theoretical H_c. Observed magnetization values are close to those observed in other methods of preparation (50 - 60 Am^2/kg) [5,6,10,17]. The value of M_r (26.58 Am^2/kg) is approximately 59% of M_s it has maximum coercivity of 6,708 Gauss for microwave calcined powder is lower than those of the literature and of the theoretical limit (7,500 Gauss) [18]. The samples calcined conventionally and microwave at 900˚C shows smooth hysteresis loop, which confirms the formation of pure strontium hexaferrite [18,19].

4. Conclusions

The effective influence of conventional and microwave on the structure and magnetic properties crystalline $SrFe_{12}O_{19}$ are discussed. The samples were subjected to two different heat treatments. From the analysis of various characterization techniques such as XRD, HR-SEM and VSM, we observe that the structure remained intact with different heating treatment process. The possibility of lowering the synthesis temperature and get a pure SrF powder, microwaves allows the reduction of particle size in the hexaferrite. The external diameters of the obtained different method of calcined $SrFe_{12}O_{19}$ particles range between 40 to 100 nm. The results indicate that calcinations method has significant effect on the saturation magnetization (M_s). These magnetic materials can potentially be used in micro/nano electronic devices, gas sensors and catalysts.

5. Acknowledgements

The authors thank SRM University for providing the facilities available in Nanotechnology center.

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